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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART VII DETERMINATION OF MANGANESE

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART VII DETERMINATION OF MANGANESE

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART VII DETERMINATION OF MANGANESE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part VII) (First Revision) was adopted by the Indian Standards Institution on 29 January 1982, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 1559-1961* which covers chemical analysis of different ferro-alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten, was first published in 1961. While reviewing this standard, the Sectional Committee felt that the standard should be revised to cover ferrosilicon, and methods of analysis of other ferro-alloys should be covered in separate standards. During preparation of the revision of IS : 1559-1961*, it was felt convenient to cover methods of chemical analysis of ferrosilicon in different parts of the standard which on publication would supersede Section 1 Ferrosilicon of IS : 1559-1961*. The committee also decided to rewrite the methods taking into consideration the latest composition specified in IS : 1110-1981†. Though calcium and manganese are not specified in IS : 1110-1981†, the determination of these elements is covered in this standard because these are generally required from the view point of determining interference of analysis in methods for determining other elements in ferrosilicon due to presence of these elements. Moreover the determination of these elements may be required for comprehensive analysis of ferrosilicon. This part covers the determination of manganese. The other parts cover as follows:

Part I Determination of silicon
Part II Determination of carbon

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*Methods of chemical analysis of ferro-alloys.

†Specification for ferrosilicon (*third revision*).

Part III Determination of sulphur
Part IV Determination of phosphorus
Part V Determination of aluminium
Part VI Determination of calcium

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part VII) covers the determination of manganese (up to 0·75 percent) in ferrosilicon.

2. SAMPLING

2.1 It shall be drawn and prepared in accordance with IS : 1472-1977†.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977‡) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF MANGANESE BY THE VOLUMETRIC (ARSENITE) METHOD

4.1 Outline of the Method — The sample is dissolved in hydrofluoric and nitric acid in a platinum crucible and the solution is fumed with sulphuric acid. The solution is cooled, diluted and boiled. From the solution, manganese is determined by the persulphate arsenite volumetric method.

5. REAGENTS

5.1 Hydrofluoric Acid — 40 percent.

5.2 Concentrated Nitric Acid — rd = 1·42 (conforming to IS : 264-1976§).

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling ferro-alloys for determination of chemical composition (*first revision*).

‡Specification for water for general laboratory use (*second revision*).

§Specification for nitric acid (*second revision*).

5.3 Dilute Sulphuric Acid — 1 : 1 (*v/v*).

5.4 Phosphoric Acid — rd = 1·75.

5.5 Silver Nitrate Solution — 17 *g/l*.

5.6 Ammonium Persulphate Solution — 25 percent (*m/v*).

5.7 Sodium Chloride Solution — 2 percent (*m/v*).

5.8 Standard Sodium Arsenite Solution (0·025 N) — Prapare the sodium arsenite solution by dissolving exactly around 1·23 g arsenious oxide (AR) in a solution of 10 g of sodium hydroxide (AR) in 30 ml of water, warming if necessary. Dilute to about 500 ml, neutralise by the addition of hydrochloric acid (1 N) to phenolphthalein end point and then add 10 g of sodium bicarbonate (AR). Dilute to 1 litre in a volumetric flask.

5.8.1 To standardise the solution take exactly 10 ml of potassium permanganate solution (0·025 N) by means of a pipette into 500-ml beaker. Add, about 5 ml of concentrated hydrochloric acid (*see* **5.9**) and boil to reduce permanganate. Add about 10 ml of concentrated sulphuric acid (*see* **5.10**) down the sides of the beaker and take the solution to fumes. Cool, dilute to about 100 ml and titrate the manganese after persulphate oxidation as described in the procedure.

Alternatively, the arsenite solution may be standardized against a sample of ferrosilicon of known manganese content.

5.9 Concentrated Hydrochloric Acid — rd = 1·16 (conforming to IS : 265-1976*).

5.10 Concentrated Sulphuric Acid — rd = 1·84 (conforming to IS : 266-1977†) .

6. PROCEDURE

6.1 Weigh exactly around 1 g of the dried sample in a 50-ml tall form platinum crucible. Add about 20 ml of hydrofluoric acid and cautiously add concentrated nitric acid dropwise until the vigorous reaction has subsided.

6.2 Add 2 ml of dilute sulphuric acid and evaporate to fumes of sulphur trioxide. Cool the crucible, add 25 ml of water and transfer the contents to 250-ml beaker. Cool and make up to 250 ml in a volumetric flask.

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*Specification for hydrochloric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

6.3 Pipette out exactly 50 ml of the solution into a 500-ml beaker, add 5 ml of phosphoric acid and dilute to 100 ml. Keep the solution on a hot-plate and boil. To the boiling solution, add about 5 ml of silver nitrate solution and 10 ml of ammonium persulphate solution. Boil the contents briskly for about 2 minutes. Cool rapidly to room temperature and add 5 ml of sodium chloride solution. Titrate immediately with standard sodium arsenite solution to a colourless end point.

6.4 Carry out a blank determination following the same procedure and using the same amount of all reagents and correct the final result for the blank value, if any.

6.5 Calculation

$$\text{Manganese, percent} = \frac{V \times N \times 0{\cdot}010\ 99 \times 100}{M}$$

where

V = volume in ml of standard sodium arsenite solution required,

N = normality of the standard sodium arsenite solution used, and

M = mass in g of the sample represented by the aliquot taken.

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